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Laser surface processing with controlled nitrogen-argon concentration levels for regulated surface life time

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ABSTRACT

Laser surface modification can be used to enhance the mechanical properties of a material, such as hardness, toughness, fatigue strength, and corrosion resistance. Surface nitriding is a widely used thermochemical method of surface modification, in which nitrogen is introduced into a metal or other material at an elevated temperature within a furnace. It is used on parts where there is a need for increased wear resistance, corrosion resistance, fatigue life, and hardness. Laser nitriding is a novel method of nitriding where the surface is heated locally by a laser, either in an atmosphere of nitrogen or with a jet of nitrogen delivered to the laser heated site. It combines the benefits of laser modification with those of nitriding. Recent work on high toughness tool steel samples has shown promising results due to the increased nitrogen gas impingement onto the laser heated region. Increased surface activity and nitrogen adsorption was achieved which resulted in a deeper and harder surface compared to conventional hardening methods. In this work, the effects of the laser power, pulse repetition frequency, and overlap percentage on laser surface treatment of 316 L SST steel samples with an argon-nitrogen jet will be presented. Resulting microstructure, phase type, microhardness, and wear resistance are presented.

1. Introduction

Laser surface modification is a beneficial processing method that can be used to enhance the mechanical properties of a material, such as hardness [1], wear resistance [2], fatigue strength [3], and corrosion resistance [4]. Surface nitriding is a thermochemical method of surface modification, in which nitrogen is incorporated into a metal or other material, at an elevated temperature. It can increase wear resistance, corrosion resistance, fatigue life, and hardness of parts [5]. In its most basic form, gas nitriding, it is performed by heat-treating the material in a pure nitrogen, or often ammonia [5], atmosphere. The process requires a long exposure time, up to 75 h. The advantages of conventional furnace gas nitriding include the improved hardness, sliding wear resistance, and corrosion resistance, that it can be performed below the phase transformation temperature, that it requires no further processing such as quenching (which could introduce warping or cracks), and that the modified layer does not alter the dimensions of the part. The main disadvantage of conventional furnace gas nitriding is its processing time. Other common types of nitriding are plasma nitriding and ion-beam nitriding, which can decrease the time and temperature needed compared to gas nitriding [6].

Laser nitriding is a novel method which combines laser surface modification with nitriding. In laser nitriding, a laser is used as the heat source, focused on the surface of the material to locally heat the surface, either in an atmosphere of nitrogen or with a jet of nitrogen delivered to the laser heated site. The technique was first reported by Katayama et. al. in 1983 [7], and has been successfully applied to many different materials and alloys, such as iron, carbon steel, stainless steel, aluminium, and titanium [5] [8–10]. Laser nitriding compares favourably to other nitriding methods, achieving comparable hardnesses and treatment depths to gas nitriding in the shortest treatment time compared to gas, plasma, or ion-beam methods [6].

Laser surface modification alone can improve the hardness and wear resistance of metal surfaces. Aqida et. al. improved the surface hardness of AISI H13 tool steel from ~300 HV to up to 1017 HV using a 1.5 kW CO_2 laser at powers of 825–1050 W, with a jet of argon delivered in line with the beam [4]. Majumdar et. al., compared the results obtained using a jet of argon, nitrogen, or a 50/50 mix of the two gases, with a 2 kW CO_2 laser on the surface of SAE 52,100 tool steel [1]. The authors found increases of microhardness ranging from ~100–200 HV for the argon jet, up to 650 HV for the nitrogen jet, and up to 700 HV for the 50/50 mixture. The wear resistance was found to improve with the hardness.

Using 100% N_2 gas may result in surface cracks and brittleness. Sun et. al. and Mridha and Baker. found the formation of the surface macro/micro-cracks in a Ti-6Al-4 V alloy laser nitrided with 100% N_2, due to the high cooling rates [11,12]. Sun et. al., reported that optimising the main laser processing parameters could reduce the residual stresses in the altered layer, and thus reduce the occurrence of surface cracks [11]. Alternatively, the application of diluted nitrogen, typically diluted with argon, can reduce cracks. However, this may also reduce the hardness achieved. Several researchers have used different ratios of argon-nitrogen gas mixtures [1,12–18]. Argon gas is typically chosen as the diluting gas because it decreases the surface tension of

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the molten material melted by the laser, allowing deeper penetration of the nitrogen in the mixture [19].

Nitriding to improve the properties of steel has possible applications in making rolling fatigue resistant gears [20], cut blades [21], bipolar plates in proton exchange membrane fuel cells [22], and biomedical applications such as surgical instruments [23]. In this work, the effect of laser processing 316 L stainless steel using an argon-nitrogen mix jet, with varied laser powers (P), pulse repetition frequencies (PRF), and percentage overlap (OV%) was investigated. The resulting samples were characterised in terms of their microstructure, phase types, microhardness, and wear.

2. Materials and methods

In this work, a computerised numerical control (CNC) CO_2 laser machine Rofin DC-015 of 1.5 kW maximum average power and a laser beam focus diameter of 0.2 mm was used. Gas could be delivered in line with the beam, using either pure argon or a mixture of 20% argon and 80% nitrogen at 0.3 mPa. A higher pressure jet may cause spreading and loss of molten material, the pressure of 0.3 mPa was found to give good results in terms of hardness with acceptably low physical material impingement. The materials used were 316 L stainless steel cylindrical pins of 10 mm diameter, the composition of which can be seen in Table 1. The cylindrical samples were processed by rotating the pin while scanning the laser linearly, to scan the laser spot over the surface of the pin in a spiral. The rotational and linear speeds could be controlled to adjust the overlap of subsequent laser spots, as well as the overlap of each line of the spiral with the previous line [24].

The laser parameters were applied according to the Box-Behnken experiment design shown in Table 2, varying the laser power (P), pulse repetition frequency (PRF), and percentage overlap (OV%) to produce 17 samples. In each case, the percentage overlap value was applied both for the overlap between consecutive laser spots and the overlap between consecutive laser tracks. Negative values of overlap correspond to the laser spots and tracks being spaced apart by a given percentage of the spotsize. The energy density threshold for melting for 316 L SST is in the range of 22–25 J/mm^2. The parameters in the DoE were chosen to be slightly above the melting threshold, to give minimal material loss via ablation. The laser pulse durations corresponding to the PRF values used are 5, 2.5, and 1.67 ms for 100, 200, and 300 Hz, respectively. One parameter set was reproduced on flat stainless steel, converting the rotational speed to linear speed and rastering back and forth in lines, using argon or nitrogen, to allow for pin-on-disc wear testing.

After processing, the microhardness, microstructure, and wear resistance were characterised. To observe the microstructure, samples were cross-sectioned, then ground and polished using a Buehler Motopol 2000. Successive grades of SiC paper of 400, 600, 800, and 1200 were applied under water flow. Final polishing was then performed using a Textmet cloth with succesive diamond and alumina suspensions of 9, 6, 3, and 0.05 μm particle size. The polished surfaces were then etched with a 5% nital etchant, made up of 95% nitric acid and 5% ethanol, by applying to the surface for 3–5 seconds with a cotton swab before rinsing. The etched surfaces were then observed by Carl Zeiss LS15 scanning electron microscope. The microhardness was measured in terms of the Vickers microhardness using a Leitz mini-load tester. The hardness indents were taken according to ASTM E18-15 with the average of five indents at specified distances from the surface recorded. A distance of five times the indent surface displacement was also used between indents in order to ensure no interference from possible strain hardening effects from previous indents. The wear was tested by the ASTM G-99 pin-on-disc standard, using a 2.5 kg load, a rotational speed of 200 RPM, a track radius of 4 mm, and a testing time of 120 min. The pins used were tungsten carbide punch pins from LinkTooling, with a hardness of 775-834 HV.

Table 1
Mass percentages for the chemical composition of the cylindrical 316 L stainless steel samples.

C	Mn	Cr	Ni	Si	P	S	Mo	N	Cu	Co	Fe
0.018	1.77	17	11.1	0.34	0.033	0.029	2.06	0.029	0.34	0.15	Bal

Table 2
Parameters and levels used for the Box-Behnken design of experiment.

	Level 1	Level 2	Level 3
Power (W)	300	400	500
PRF (Hz)	100	200	300
Overlap (%)	−20	0	20

3. Results and discussion

3.1. Altered surface hardness

The average hardness of the untreated stainless steel cylindrical samples was found to be 250–280 HV. The hardness after laser processing with the argon-nitrogen gas mixture was found to have increased significantly for a number of samples. The hardness recorded for the Box-Behnken samples laser processed with 20%Ar-80%Ni or with 100% Ar can be seen in Table 3. The highest value, of 590 HV found for sample 6 treated with the argon-nitrogen mix, is over double the untreated hardness. The average hardness for the five replicates at 400 W, 200 Hz, and 0% overlap with 20%Ar-80%Ni is 333 HV, with a 95% confidence interval of 16 HV. The improvement in the hardness had depths of up to 900 μm. In Fig. 1, a plot of hardness vs depth for sample 1 and sample 6 of the set processed with 20%Ar-80%Ni, the samples with the highest hardness at the surface, is shown. The hardness decreases with depth, with sample 6 reaching the initial bulk hardness at ∼900 μm below the surface, and remaining >500 HV for over 400 μm. These depths are significantly above those noted for plasma nitriding of 316 L SST, where Biehler et. al. for example measured nitriding depths of ≤ 7.2 μm for plasma nitriding with 300 Pa pressure [25]. However, these authors achieved surface hardness of up to 1662 HV.

The data from Table 3 is shown as response surface graphs in Fig. 2 to illustrate the effect of the laser processing parameters on the resulting micro-hardness of the 316 L stainless steel cylindrical samples. Fig. 2 (b) indicates a strong direct proportionality between the PRF and the resulting hardness at the surface. This agrees with trends reported in the literature [5]. This relationship can be explained by the higher PRF leading to a shorter residence time and therefore faster solidification which is known to result in a harder surface material. Achieving the

Table 3
Laser parameters and resulting microhardness for the laser processed SST pin samples.

Sample	Power (W)	PRF (Hz)	OV%	20%Ar-80%Ni Microhardness (HV)	100%Ar Microhardness (HV)
1	300	200	−20	549	342
2	400	200	0	301	304
3	400	300	−20	446	338
4	400	200	0	342	304
5	500	200	−20	363	324
6	400	300	20	590	347
7	500	300	0	331	346
8	400	200	0	339	304
9	400	200	0	345	304
10	400	100	20	307	313
11	400	100	−20	462	313
12	300	300	0	300	344
13	300	200	20	326	343
14	500	100	0	315	343
15	500	200	20	286	243
16	300	100	0	310	313
17	400	200	0	338	304